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Internal stress induced texture in Ni-Mn-Ga based glass-covered microwires

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We have studied magnetic and structural properties of the composite microwires consisted of the metallic core and the outer glass shell. Nominal chemical composition of the core was $\text{Ni}_{49.5}\text{Mn}_{25.4}\text{Ga}_{25.1}$, its diameter was $13.2\ \mu\text{m}$, and the total diameter of the glass-covered microwires was $26.4\ \mu\text{m}$. We have found out that at room temperature the core of the as-cast microwires was composed by two phases with tetragonal I4/mmm and cubic Fm3m crystal structures, but annealing rendered it single phase. Measurements of the magnetic properties have demonstrated substantial growth of the magnetic anisotropy with cooling, which we have attributed to the phase transition from the room-temperature austenitic to the low-temperature martensitic state. Magnetic easy axis was found to be perpendicular to the axis of the microwires at low temperatures. We believe that it is a result of the crystallographic texture induced in the martensite by high internal stress characteristic of the glass-covered magnetic microwires. Though rearrangement of the martensitic microstructure under external pressure was previously observed in the single crystal Ni_2MnGa samples, in composite materials this effect is new and can be potentially useful for the applications. © 2013 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4822168>]

I. INTRODUCTION

Remarkable properties of Heusler alloys such as shape-memory effect, large field-induced strain, half-metallic behavior, giant magnetocaloric effect, exchange bias, and other unique features are known for a long time.^{1,2} However, these materials are still rarely used in the applications. One of the problems hampering wider use of Heusler alloys is their brittleness. It was found to be difficult to produce thin wires, ribbons, and other small structures³ out of Heusler alloys. Preparation of the composites containing Heusler alloys was found to be a way of circumventing this problem and became a hot topic in the development of this family of the functional materials.^{4,5}

Thin composite wires consisted of the metallic core and the outer glass shell can be fabricated using so-called Taylor-Ulitovsky technique. It allows producing of few km long pieces with typical diameters of the metallic core from 1 to $30\ \mu\text{m}$ and with the thickness of the glass coating between 0.5 and $20\ \mu\text{m}$.^{6–11} Glass-covered microwires with amorphous metallic core made out of the magnetic Co-based alloys demonstrate giant magnetoimpedance effect. Now they are used in the commercially available magnetic field sensors.^{10,11} Recently Taylor-Ulitovsky technique has been used for preparation of the microwires with granular structure from the (Co, Fe, Ni)-(Cu) alloys,^{12,13} microwires exhibiting mixed amorphous-crystalline structure,¹⁴ microwires with enhanced magnetocaloric effect,¹⁵ and microwires from Ni-Mn-Ga Heusler alloys.¹⁶

Non-stoichiometric Ni-Mn-Ga alloy is a generic term for the alloys derived from the stoichiometric $\text{Ni}_{50}\text{Mn}_{25}\text{Ga}_{25}$ composition by variation of the relative amount of constitutive elements. Many interesting properties of this family of Heusler alloys result from the strong coupling between the magnetic and structural order. It was found that in the wide range of the concentrations Ni-Mn-Ga alloys have $L2_1$ cubic crystal structure in the high-temperature austenitic phase and display martensitic phase transition on cooling. Although few different complex crystal structures were observed in the martensitic phase, schematically martensitic transformation can be represented by shrinking along one of the edges of the parent cubic structure and expansion along two other edges. It results in pseudo-tetragonal (10M modulated) or pseudo-orthorhombic (14M modulated) structures with $c/a < 1$ or in non-modulated tetragonal structure with $c/a > 1$.^{1,3,17} C-axis is the easy-magnetization direction of both pseudo-tetragonal and pseudo-orthorhombic lattices and the hard-magnetization direction of the non-modulated tetragonal one.¹⁷

Since phase transition to the martensitic state gives rise to appearance of the cells with c-axes parallel to the each of three equivalent directions of the parent cubic structure, it leads to the breakdown of the initially single-crystalline grains into crystallographic domains (crystallographic twins). External stress induces rearrangement of this domain structure. For example, compressive stress makes energetically more favorable twins with short axis parallel to the direction of the stress and provokes their growth in expense of the less favorable ones. Stress of few MPa was found to be sufficient to restore single-domain crystallographic structure.^{1,17}

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This paper presents details of the preparation and magnetic properties of the glass-covered microwires whose metallic core was made out of the near-stoichiometric Ni_2MnGa alloy. Besides characterizing crystal structure and measuring magnetization as a function of temperature and field we have tried to reveal the effect of the internal stress on the magnetic properties of these samples.

Internal stress is characteristic of glass-covered microwires. It appears because of the difference in the thermal expansion of the metallic and glass parts of the samples. Its value depends on the ratio of the diameters of the core and the whole microwire and can reach few GPa.^{7,8} In the case of the core made of Heusler alloy we expected to see some stress-induced crystalline texture and respective anisotropy of the magnetic properties.

II. EXPERIMENTAL DETAILS

Long (few hundred meters) continuous glass-covered microwire with diameter of the internal metallic core of $13.2\ \mu\text{m}$ and the total diameter of $26.4\ \mu\text{m}$ has been produced in one cycle of Taylor-Ulitovsky process.⁵ The ingot of $\text{Ni}_{49.5}\text{Mn}_{25.4}\text{Ga}_{25.1}$ composition used in the process was prepared by arc melting of 2 N pure elements.

Samples for magnetic measurements have been prepared from 5 mm long pieces of the microwire fastened in the bunches. Magnetic properties were investigated using both Lake Shore (7400 System) vibrating-sample magnetometer (VSM) and physical properties measurements system (PPMS). If other is not specified, magnetic characteristics were measured with magnetic field parallel to the axes of microwires in the bunch.

Conventional Siemens X-ray diffractometer was used to carry out a crystal structure characterization. Energy dispersive X-ray composition analysis (EDX) was performed for detection of the microwire's metallic core composition.

Annealing has been performed in electrical muffle furnace in air. Glass coating protected the core from the oxidation. The sample was heated up to 790 K, kept for 30 min, and then cooled down to the room temperature at 2 K/min rate.

III. EXPERIMENTAL RESULTS

SEM image presented in Fig. 1 shows a circular cross section of the glass-covered microwire. Diameter of the metallic nucleus is $13.2\ \mu\text{m}$, and the total diameter of the microwire is $26.4\ \mu\text{m}$.

X-ray characterization performed at room temperature revealed coexistence of the cubic austenite (space group $\text{Fm}\bar{3}\text{m}$, lattice parameter $a = 0.582\ \text{nm}$) and tetragonal martensite (I4/mmm , $a = 0.387\ \text{nm}$, $c = 0.428\ \text{nm}$) phases in the as-cast sample. Annealed sample was single-phase with crystal structure typical for the cubic austenite ($\text{Fm}\bar{3}\text{m}$ space group, lattice parameter $a = 0.582\ \text{nm}$). The same structures have been observed in the glass-covered microwires with similar diameters made out of the stoichiometric Ni_2MnGa . However, the crystal structure of the thicker microwires was tetragonal at room temperature both in the as-cast and annealed samples.¹⁶

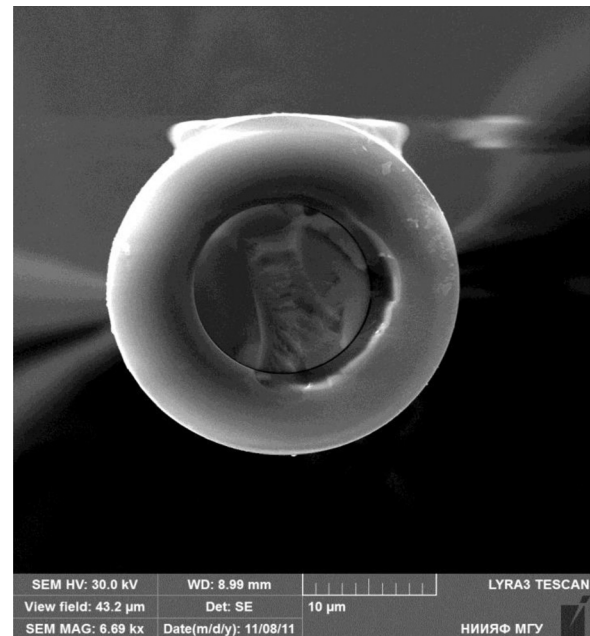


FIG. 1. SEM image of the single microwire cross section.

The EDX characterization performed at different points of the metallic nucleus after the glass removal yielded chemical composition of $\text{Ni}_{62}\text{Mn}_{13}\text{Ga}_{25}$. Remarkable difference between the nominal and observed compositions may be attributed to the Mn evaporation during the fabrication or to the chemical reaction with the acid used to remove the glass.

Fig. 2 shows temperature dependences of the magnetic moment, M , for as-cast and annealed samples measured in the magnetic field of 1.2 kOe using PPMS. To compare these curves we have normalized both dependences taking as a reference the magnetic moment at the lowest temperature, M_0 . Magnetic ordering appears in the as-cast microwire around 175 K. The sample seems to contain the second magnetic phase which orders at low temperature giving rise to the fast increase of the magnetic moment below 30 K. Meanwhile we have observed common ferromagnetic behaviour for the

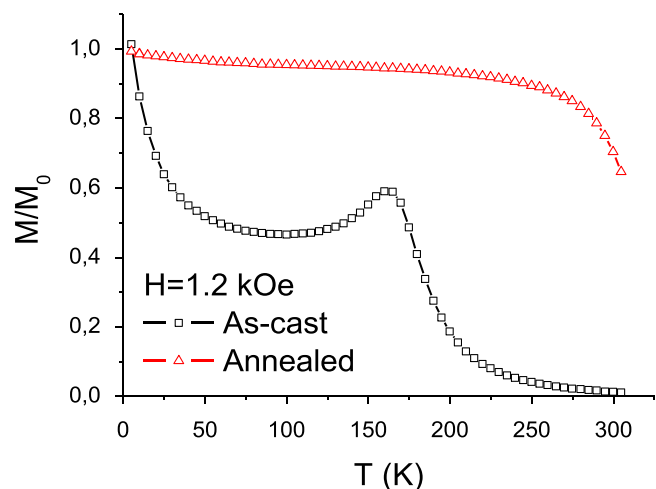


FIG. 2. Magnetic moment of the as-prepared and annealed samples measured in the magnetic field of $H = 1.2\ \text{kOe}$ versus temperature (M_0 —is the magnetic moment at the lowest temperature).

annealed sample. There is no any evidence that it contains second magnetic phase. The Curie temperature (T_C) estimated as an inflection point on the $M(T)$ dependence was found to be about 325 K.

We believe that this drastic change of the magnetic properties after annealing is concerned with the substantial inhomogeneity of the crystal structure and non-uniform distribution of different elements within the core of the as-cast microwires. Even in the samples prepared by conventional methods Ga/Mn atomic disorder was observed in the as-cast state.^{18,19} The microwires made by Taylor-Ulitovsky process are quenched from melt faster than 10^5 K/s,⁶ which easily allows existence of the metastable phases.

Temperature dependence of the magnetic moment of the annealed sample measured using VSM technique is shown in Fig. 3. We used field-cooled, field-heated regime (FC-FH) for the measurements performed at 1 kOe. The $M(T)$ dependence measured at 1.5 kOe has been obtained in zero-field-cooled, field-cooled regime (ZFC-FC). Near T_C both curves exhibit temperature hysteresis in the range from 260 to 350 K. The width of the temperature hysteresis depends on the rate of the heating/cooling. In the range of 1–3 K/min rates the hysteresis changes from 4 to 6 K, becoming narrower for slower measurements. Zero-field-cooled branch of the ZFC-FC curve shows that the projection of the magnetic moment parallel to the field grows with heating in the range of temperatures from 80 to 200 K when pre-cooled sample is exposed to the field of 1.5 kOe.

Hysteresis loops measured for the annealed sample are presented in Fig. 4 (shown only parts corresponded to the increasing of the magnetic field). Magnetic anisotropy and saturation field clearly grow with decreasing of the temperature. Magnetic field of 1.5 kOe is not enough to saturate the sample in the range of temperatures from 80 to 200 K. According to this figure, values of the magnetic moment taken at $H = 1.5$ kOe grow up to 200 K although the saturation magnetic moment monotonically decreases with temperature. It explains growth of the ZFC branch of the curve in Fig. 3.

Indeed, saturation fields in the range of few kOe are not typical for the austenitic phase of the Ni-Mn-Ga alloys

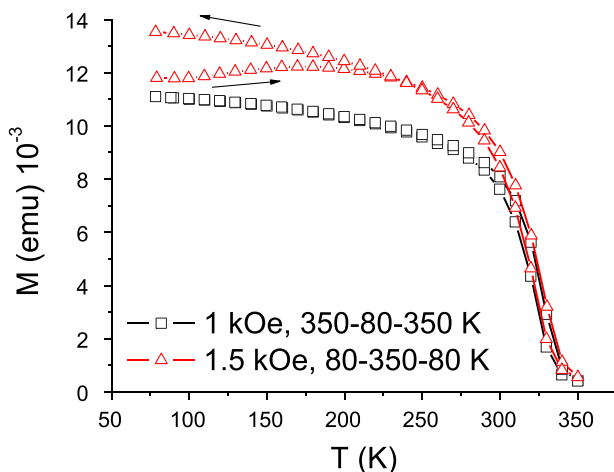


FIG. 3. Magnetic moment of the annealed sample measured in the magnetic fields of 1 and 1.5 kOe versus temperature (heating and cooling processes shown by the arrows).

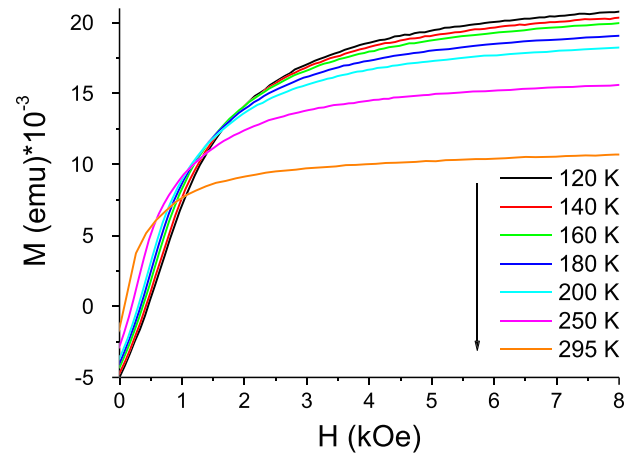


FIG. 4. Hysteresis loops of the annealed sample (shown only parts corresponded to the growth of the field); field is parallel to the axes of the microwires in the bunch.

but characteristic of the more anisotropic martensite. For example, for $\text{Ni}_{51.3}\text{Mn}_{24.0}\text{Ga}_{24.7}$ (temperature of the martensitic phase transition (T_M) is 263 K, $T_C = 358$ K) magnetic field of 700 Oe was enough to saturate the austenite, but the martensite demonstrated saturation magnetic fields from 2 to 9 kOe.²⁰ In our case the saturation magnetic field at 120 K is around 2 kOe. Therefore we conclude that at least at this temperature sample has converted from the austenite to the martensite.

Hysteresis loops measured along and perpendicular to the axes of the microwires revealed that the coercive force for the transverse direction is higher than for the axial one at temperatures lower than 295 K—at higher temperatures coercive forces are the same for both directions. Temperature dependencies of the coercive force are shown in Fig. 5.

Hysteresis loops measured at 180 K for both orientations of magnetic field are presented in Fig. 6. The experimental curve corresponded to the measurements in the transverse magnetic field is more flat but should be corrected taking into account the demagnetizing field. Internal field is given by equation $H = H_{ex} - 2\pi I$, where I is a magnetization and H_{ex} is a value of the applied magnetic field. This correction takes

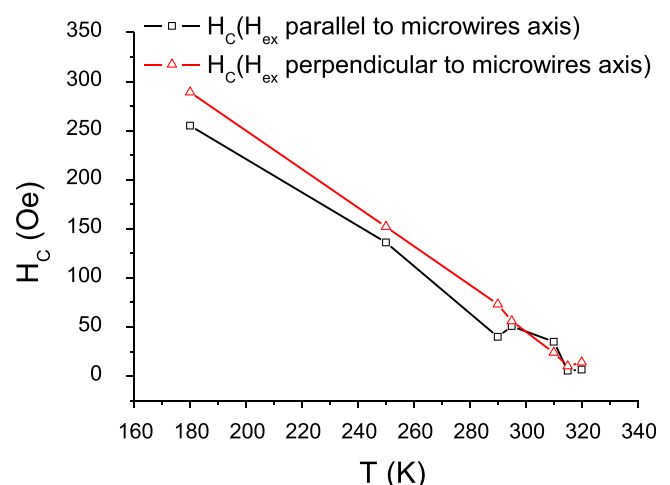


FIG. 5. Coercive force obtained from the hysteresis loops measured parallel and perpendicular to the axes of the microwires.

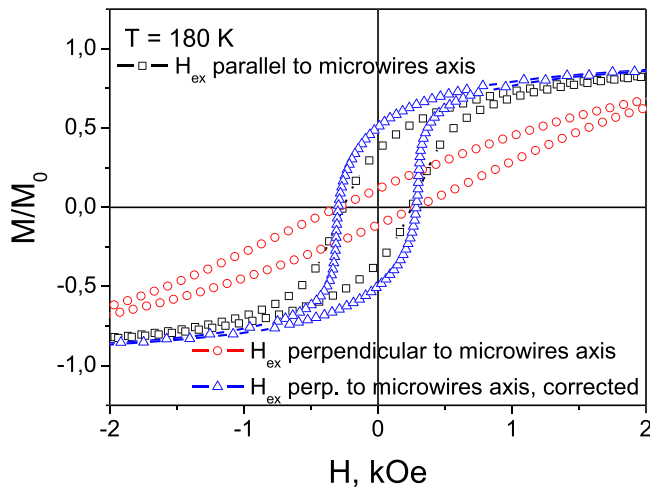


FIG. 6. Hysteresis loops measured parallel and perpendicular to the axes of the microwires at 180 K.

values of the magnetization of the metallic nucleus itself meanwhile the measurements give us the value of the magnetic moment of the whole sample, including the glass shell and the sample holder. The accurate separation of the different contributions is not simple. Instead we have found out that the slope of the curves will be similar for both directions if the saturation magnetization of the metallic nucleus is only 300 emu/cm^3 . It is two times lower than the saturation magnetization of $\text{Ni}_{51.3}\text{Mn}_{24.0}\text{Ga}_{24.7}$ ($I_S = 600 \text{ emu/cm}^3$).²⁰ Since magnetic moment of Ni-Mn-Ga Heusler alloys is mainly defined by the Mn concentration, our sample should have only 12 at. % of Mn to match the I_S value of 300 emu/cm^3 . This concentration of Mn is even lower than obtained by the EDX measurements. The real content of Mn in our sample is higher. It means that the hysteresis loop corrected for the demagnetizing field is more flat for the longitudinal direction, and the easy magnetization direction is perpendicular to the axis of microwire.

These findings are compatible with the distribution of the internal stress in the glass-covered microwires. At least in the central part of the metallic nucleus the axial tensile stress is bigger than the other components of the stress.^{7,21,22} Since the martensite with near stoichiometric composition has pseudo-orthorhombic (14 M modulated) or pseudo-tetragonal (10 M modulated) structure with $c/a < 1$ and c -axis is the easy magnetisation direction,^{1,3,17} this stress should cause a texture with preferential orientation of the shorter c -axis perpendicular to the largest stresses component. Therefore the easy magnetization direction perpendicular to the microwire axis must be induced. Lower values of the coercive force observed for the longitudinal loops corroborate this conclusion. Furthermore, merging of the coercive forces above 295 K confirms our observation that at room temperature sample has austenite structure because stress induces no texture in the austenite with cubic crystal structure.

IV. DISCUSSION

Martensitic phase transitions in bulk Ni_2MnGa -type Heusler alloys are usually accompanied by pronounced thermal hysteresis of the magnetization. In our case difference

between the heating and the cooling branches of the $M(T)$ curve is not very big, but the region of the hysteresis is wide. It spreads from 260 to 350 K. We believe that this behavior is a result of the variation of the temperature of the martensitic phase transition within the sample. Most probably it is concerned with strong change of the internal stress along the radius of the microwires. Inhomogeneous chemical composition could also be a reason, but it should blur the ferromagnetic-paramagnetic phase transition which is quite sharp even in rather high fields.

If chemical composition of the microwires measured using EDX after the chemical removing of the glass is the same as of the virgin glass-covered microwires, then measured magnetic properties contradict known magnetic phase diagram of Ni_2MnGa -type Heusler alloys.

Concentration-weighted average e/a corresponded to the nominal and EDX-measured chemical composition of the core is 7.48 and 7.86, respectively. These values have been calculated taking into account the total numbers of s and d electrons for Ni and Mn, and s and p electrons for Ga (i.e., 10, 7, and 3 electrons, respectively).

The phase diagram¹ shows that T_C decreases and T_M increases with e/a until they merge at $e/a \sim 7.7$. Then temperature of the combined phase transition grows with e/a . The lowest observed value of the Curie temperature was around 320 K. Similar phase diagram has been built for the series $\text{Ni}_{50-x}\text{Mn}_{25+x}\text{Ga}_{25}$,²³ the lowest Curie temperature was reported to be 325 K for $\text{Ni}_{54}\text{Mn}_{21}\text{Ga}_{25}$. According to this phase diagram, for the Ni-Mn-Ga alloy with $e/a \approx 7.86$ we must observe $T_C \approx 400 \text{ K}$. However we have experimentally observed $T_C \approx 325 \text{ K}$ which agrees well with e/a of 7.48. We have concluded that either EDX method yielded composition with overestimated Ni ratio or chemical composition changed due to removing of the glass with the HF acid. We believe that the chemical composition of the core of the studied glass-covered microwires is close to the nominal value.

V. CONCLUSIONS

We have studied magnetic and structural properties of composite microwires consisted of the metallic core and the outer glass shell. Nominal chemical composition of the core was $\text{Ni}_{49.5}\text{Mn}_{25.4}\text{Ga}_{25.1}$, its diameter was $13.2 \mu\text{m}$, and the total diameter of the glass-covered microwires was $26.4 \mu\text{m}$. We have found out that at room temperature the core of the as-cast microwires was composed by two phases with tetragonal $I4/mmm$ and cubic $Fm3m$ crystal structures but annealing rendered it single phase. Measurements of the magnetic properties have demonstrated substantial growth of the magnetic anisotropy with cooling, which we have attributed to the phase transition from the room-temperature austenitic to the low-temperature martensitic state. Magnetic easy axis was found to be perpendicular to the axis of the microwires at low temperatures. We believe that it is a result of the crystallographic texture induced in the martensite by high internal stress characteristic of the glass-covered magnetic microwires. Though rearrangement of the martensitic microstructure under external pressure was previously observed in single crystal Ni_2MnGa

samples, in composite materials this effect is new and can be potentially useful for the applications.

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